



LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

UCRL-TR-230829

Engineered Aerosol Production for Laboratory Scale Chemical / Biological Test and Evaluation

G. M. Dougherty, D. R. Hadley, P. R. O'Connor, J.
R. Bottiger

May 9, 2007

Disclaimer

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

UCRL-#####-#####



Engineered Aerosol Production for Laboratory Scale Chemical / Biological Test and Evaluation

DTRA – JSTO Project CA06TAS446

Final Report

Prepared By:

George M. Dougherty (Principal Investigator)

Dean R. Hadley

Patrick R. O'Connor

Lawrence Livermore National Laboratory

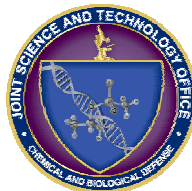
Jerold R. Bottiger

U.S. Army Edgewood Chemical Biological Center

for

Defense Threat Reduction Agency – Joint Science and Technology Office

9 May 2007



This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

Contents

1. Introduction
2. Project Overview
3. Results
 - 3.1. Core System Development
 - 3.2. Software Development
 - 3.3. Droplet Conditioning Module
 - 3.4. Additional Hardware Components
 - 3.5. Aerosol Testing
 - 3.5.1. Size distribution measurements
 - 3.5.2. Particle size control
 - 3.5.3. Biological slurries and spore simulants
 - 3.5.4. Programmed sequence operation
4. Programmatics
5. Summary

APPENDIX

System software and operation

1. Introduction

As chemical and biological detection instrumentation has become more sophisticated and higher in performance, there has been a corresponding need for more sophisticated and advanced test and evaluation capabilities. One specific need is for advanced aerosol generation technology that can enable the next generation of realistic, complex, and dynamic simulations of real-world aerosol environments in the testing laboratory. Current devices for aerosol generation, such as glass nebulizers, have remained relatively unchanged for decades.

The advent of electronically controlled inkjet technology for printing and imaging has created exciting new possibilities for aerosol generation that is much more controllable, dynamic, and reproducible than any that has existed before. The potential for inkjet-based aerosol generation was first demonstrated by the U.S. Army's Edgewood Chemical Biological Center (ECBC). The Inkjet Aerosol Generator, or IJAG, mated the 12-nozzle thermal inkjet head from a desktop inkjet printer with a heated dessication tube and associated hardware, as shown in Fig. 1. Custom software controls the device operation. Instead of striking paper to form an image, the droplets generated by the nozzles pass into the dessication tube where they are rapidly dried. From the end of the tube emerge dry aerosol particles consisting of the nonvolatile dissolved substances from the liquid solution being printed.



Fig. 1. The Inkjet Aerosol Generator (IJAG) developed at ECBC. The inkjet printhead is located at the top of the white dessication tube. The large box houses power supplies, and monitors airflow for the satellite droplet rejection feature.

Such IJAG units have proven very useful for testing biodetection instrumentation, because when the aerosol output is directed into the inlet of a biodetector, digital control allows for the number of particles being introduced to the detector to be known exactly. In addition, the particles are very uniform, and the generation rate can be varied over many orders of magnitude, all the way down to single-particle mode.

However, the IJAG unit has some shortcomings that make it impractical for use in generating aerosols for a dynamic environmental test chamber. First, the maximum particle generation rate for the 12-nozzle array is about 48,000 per second, too low to fill a test chamber in a reasonable time. The IJAG uses a single head and a single solution, so that the generation of complex aerosol mixtures requires separate units. And the thermal inkjet mechanism can lead to fouling and clogging when biological slurries and other complex liquids are used in place of ink.

Therefore, the current project was undertaken to scale up the inkjet aerosol technology to high generation rates, multi-component aerosol capability, and a non-thermal piezoelectric inkjet mechanism that is more robust for use with a wide range of liquids. Lawrence Livermore National Laboratory (LLNL) and ECBC combined their capabilities to carry out the development work.

The new system, known as the Digital Aerosol Generator (DAG), consists of the basic components shown in Fig. 2. Large scale inkjet array printheads, developed commercially for industrial printing applications, generate inkjet droplets using a non-thermal piezoelectric mechanism. Up to four such printheads are used, in combination with dessication modules to dry the liquid droplets down to aerosol size. Fluidic subsystems are used to supply each printhead with liquid solution, and a computer with graphical user interface (GUI) control software is used to manage the operation of the overall system.

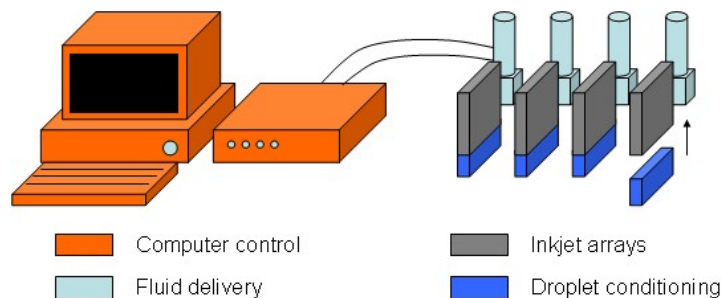


Fig. 2. Conceptual diagram of the Digital Aerosol Generator (DAG) system.

The key enabling components are the commercial off-the-shelf (COTS) inkjet array printheads. Were these not available, the development of the system would not be practical. The printheads used for the DAG have recently been introduced by Konica Minolta (Konica Minolta Technology USA, Inc., Fremont, CA). The KM256 printhead and KM512 printheads are shown in Fig. 3. The KM256, with 256 independent nozzles, is designed for printing with aqueous solutions, while the KM512 is for use with non-aqueous solvents.

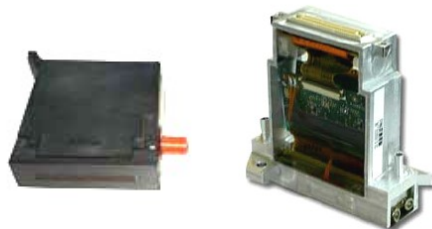


Fig. 3. Images of the COTS inkjet array printheads used in the DAG. The KM256, at left, is for use with aqueous liquids and was the standard printhead employed throughout the development program. The KM512, at right, is for liquids based on non-aqueous solvents. With some modifications, the DAG has the capability to accept either type of printhead.

2. Project Overview

The development and testing of the Digital Aerosol Generator (DAG) prototype was performed as a 12-month project, running from April 2006 to April 2007, with a total budget of \$541K. During final budget negotiations it was determined that, to reduce the project cost, the project scope would include the design of a system capable of operation with four printheads, but testing of only single-printhead operation. The project effort was divided into the following tasks:

Task 1 – Core Inkjet System Buildup (performed at LLNL)

The core inkjet system consists of the printhead(s), liquid supply system, and basic electronic / computer software control. It does not include the subsystems for droplet conditioning (dessication), or much of the final software functionality. The early construction and testing of the core system allowed for an early verification of the

principle of operation of the DAG. Since a great deal of the engineering challenge of the project would lie in the integration of custom hardware and software with the COTS printheads and associated electronics and their embedded firmware, the core system buildup was expected to be a substantial task. Once basic operations were established, characterization of the droplet generation performance of the printheads with different solutions allowed protocols to be determined for the subsequent operation of the system for aerosol generation.

Task 2 – Droplet Conditioning (performed at ECBC)

The droplet conditioning module(s) accept the liquid droplets produced by the inkjet printheads and dry them to yield aerosol particles. In the case of the IJAG, a backflow of air within the module also served to eliminate small “satellite” droplets sometimes produced along with the main inkjet droplets, and a similar capability was developed for the DAG. This task included the design and fabrication of the modules, some local testing at ECBC, and the shipping of the hardware to LLNL for integration with the core inkjet system.

Task 3 – Development Testing (performed at LLNL)

Once the droplet conditioning hardware was mated with the core inkjet system and the software development was sufficiently advanced, a series of tests were performed to characterize the operation and performance of the full system. These included aerosol chamber tests, instrumented with an aerosol particle size analyzer, to verify the ability of the DAG to generate well-controlled particles from different solutions including biological particle (i.e. bacterial spore) and simulant slurries. Tests would also verify the capability of the system to generate aerosols in automated mode, using pre-programmed time varying generation rate profiles.

3. Results

3.1 Core System Development

The core system was built at LLNL, combining the KM256 aqueous heads and EB-100 electronics unit obtained from Konica Minolta with custom fixturing, variable-height gravity-feed fluidics, and a computer with control software. A photograph of the printheads and associated fixturing is shown in Fig. 4. The earliest droplet generation tests were conducted using demonstration software provided by Konica Minolta, and later tests were conducted with the team’s custom built LabView code, as it was developed (see section 3.2 below).

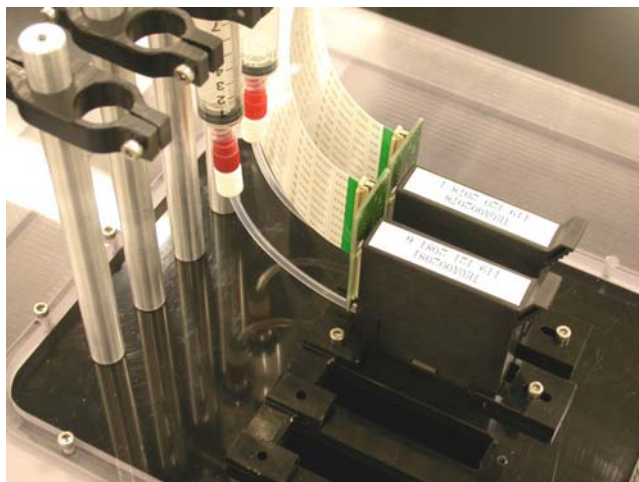


Fig. 4. Configuration of the printheads, liquid supply fluidics, and fixturing for the core system. The system is shown with two printheads installed. The control computer and EB-100 electronics unit are not visible.

Apart from various forms of operational testing to verify mechanical and electrical functions of the core system, the important testing involved stroboscopic imaging characterization of the droplet formation dynamics. By tapping the trigger pulses generated by the electronics unit, pulse-gated stroboscopic video of droplets emerging from the printhead was captured for a range of different aqueous liquid viscosities and waveform parameters. This allowed direct observation of the droplet formation, and assessment of the quality of droplet formation at each condition.

Several droplet formation regimes were observed, and these were reproducible with liquids of different viscosities, though the waveform pulse widths at which they occur were observed to vary with viscosity. At the shortest pulse widths, there was no droplet generation, and if the width were increased slightly, a regime of sporadic generation was sometimes observed, wherein individual nozzles produced droplets at uneven intervals, and the generation rate was much lower than expected. Increasing the pulse widths still more brought about the optimal regime of single droplet formation. This is the desired regime both for inkjet image printing and for most aerosol generation. At longer pulse widths the formation of satellite droplets could be observed, small secondary droplets that lag the primary droplets. These could be transient, such that the satellites catch up with and merge with the primary droplets, or permanent, as seen at the longest pulse widths. Images of droplets formed in two regimes, single droplet and satellite droplet, are shown in Fig. 5.

The pulse widths specified refer to the “D” rise time that automatically sets the other two pulse shape parameters in the “DRC” profile control used by the EB-100 unit to drive the printhead nozzles. In the standard DAG operating mode, the “R” parameter is fixed at twice the “D” time, and the “C” parameter is disabled.

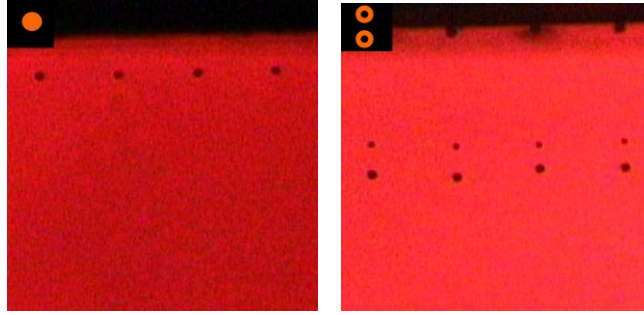


Fig. 5. Stroboscopic video images of liquid droplets formed in two different regimes. At left, the combination of waveform pulse length and solution viscosity results in single droplets. At right, different conditions result in the formation of satellite droplets.

The recommended viscosity range for the KM256 head, as provided by the manufacturer, is 3-12 centipoise (cP). Tests were carried out at 1.0, 2.9, 6.0, and 17.7 cP, using solutions of water and glycerine in controlled ratios. The test results are shown in Fig. 6. Optimum pulse widths are shorter for solutions of lesser viscosity, the optimal for pure water (1 cP) lying near 2.5 μ s. The ranges of pulse widths that result in single droplets, as well as the length of the optimal pulse widths themselves, increase with increasing viscosity. At the highest viscosity of 17.7 cP, single droplets were observed at pulse widths of 6 and 7 μ s.

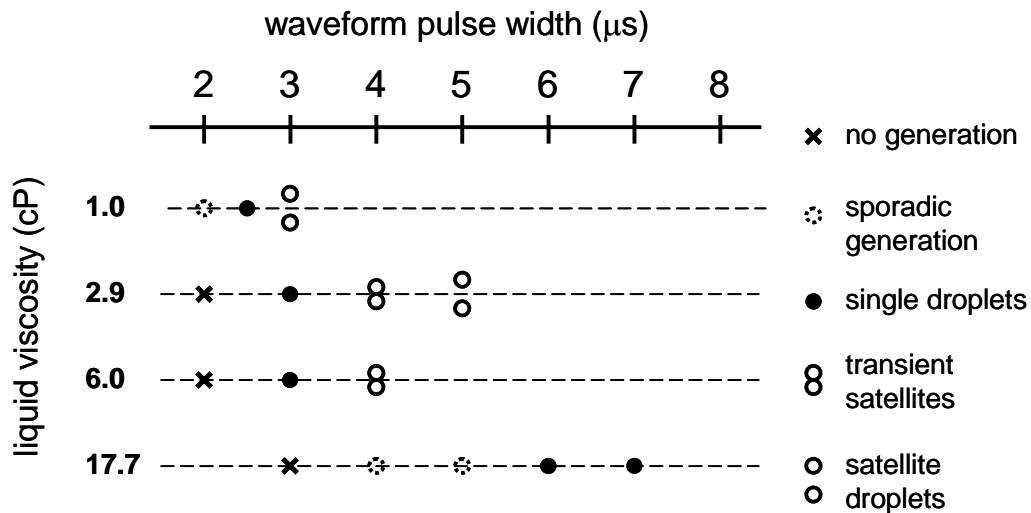


Fig. 6. A chart summarizing the results of droplet dynamics tests. The waveform pulse widths refer to the “D” rise time, which automatically dictates the other waveform parameters in the standard DAG operating mode. Note that by controlling the pulse width, single droplets can be generated without need of the aerodynamic sorting used in the IJAG.

Stroboscopic imaging tests were also done to verify the effective generation of droplets at different droplet generation frequencies. No significant change in droplet shape or regime was observed with frequency. Images clearly show the repeatable generation of identical single droplets at frequencies as high as 15 kHz. A stroboscopic image of high rate generation is shown in Fig. 7.

The results of the stroboscopic imaging tests verify that inkjet droplets can be produced from aqueous solutions with a wide range of viscosities, and that the droplet dynamics can be tuned by adjusting the electronic pulse width sent to the print nozzles. This level of electronic tuning means that air backflow, as used in the IJAG, is not necessary to ensure a monodisperse population of droplets in this system.

It was verified that printheads may be effectively cleaned by immersing the nozzles in a shallow (a few mm) layer of purified water in an ultrasonic cleaning bath, and backflushing the printheads using a syringe attached to the fluid feed port.

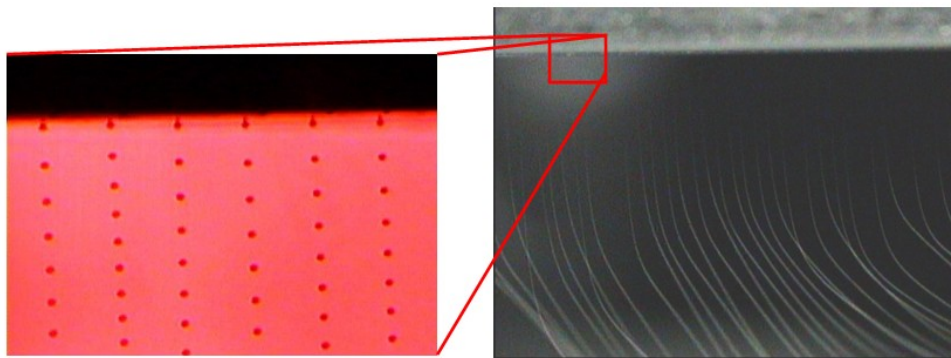


Fig. 7. A standard black and white video zoom image of droplets emerging from a KM256 printhead, and a stroboscopic video microscopy image of droplets emerging from the printhead at a 10 kHz print frequency (for a total of 2.56 million droplets per second from the printhead). Nozzle spacing is 141 μm , and the field of view of the image on the right is approximately 2 cm.

3.2. Software Development

The development of a full-featured software application to allow a user to conveniently operate the Digital Aerosol Generator comprised a large fraction of the development work during the project. The COTS printheads were acquired with a dedicated Konica Minolta printhead driver electronics subsystem, designated the EB-100, with embedded firmware optimized for image printing applications. The EB-100 accepts serial commands from the control computer and converts them into the series of digital pulses required to print an image. While this allowed the development team to avoid dealing directly with bit-level control over very complex printing dynamics and data buses, the specialized imaging functions of the firmware had to be adapted and, in some cases, circumvented to allow the types of control more appropriate for the aerosol generation application.

The custom software is built in LabView 8.0 (National Instruments), and makes extensive calls to the digital linked library of driver commands for the EB-100 unit. The earliest iterations of the software included only the most basic functions, and additional functionality was added in the form of version upgrades over the course of the project. Because the COTS hardware and firmware was itself very new (the developer's manual for the KM256 printheads had not been released at the time we began work) a lack of documentation made the software interface work especially challenging. Nonetheless,

the final version of the control software contains a wide range of powerful features and is very user-friendly.

Over the course of the project, features were added in stepwise fashion to the basic print operations, including:

- Control of the generation frequency (droplet generation rate)
- Automatic selection of appropriate waveform pulse widths based upon the viscosity of the liquid solution to be aerosolized
- Control over printhead filling, and closed-loop control over liquid supply pressure during printing
- User-friendly definition of pre-programmed generation rate profiles to allow automated execution of time-varying test conditions
- Selection for operations with 1 to 4 printheads

An image of the front panel of the graphical user interface (GUI) is shown in Fig. 8.

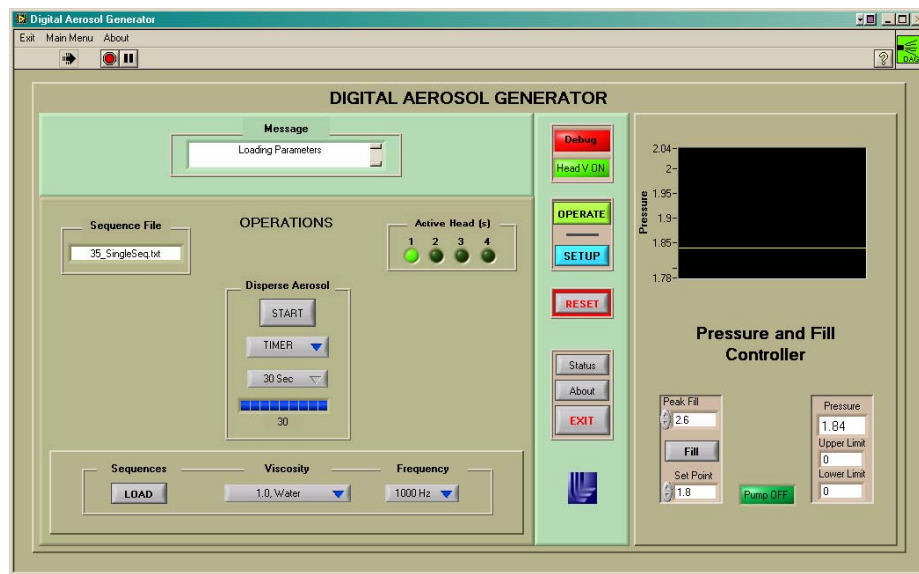


Fig. 8. The front panel of the Graphical User Interface (GUI) control software for the Digital Aerosol Generator. The left side provides control of basic aerosol generation operations, and the right provides control of the closed-loop liquid pressure supply system.

3.3. Droplet Conditioning Module

The KM 256 printhead produces droplets nominally 30 microns in diameter that will dry down to residue particles of a few microns diameter, the final size depending on the concentration of the material mixed with water and fed to the printhead. The rate at which droplets are made is controlled and known; to insure that the aerosol generating system overall has a quantitative output, particle losses must be kept at a minimum. Particles the size of the original droplets, 30 microns, are too heavy to remain aerosolized

and are easily lost through inertial processes, and so we arrange to dry the droplets quickly (< 2 seconds) by firing the printheads into a stream of pre-heated air.

The heart of the droplet conditioning (drying) module is a hot air duct, 3 ½ inches wide and 1 ½ inches high (inside dimensions) and 8 inches long. It is constructed from ½ inch thick plates of Teflon, which was chosen for its combination of low thermal conductivity, high working temperature (up to 500 °F) and machinability. The inside walls are lined with aluminum sheet to help equalize the temperature along its length, and a layer of ceramic tape between the aluminum and the Teflon retards heat transfer into the plastic. An image of a drying module is shown in Fig. 9, along with some of the auxiliary components used in development tests. Fig. 10 shows a closeup of the printhead and exit region of the module.

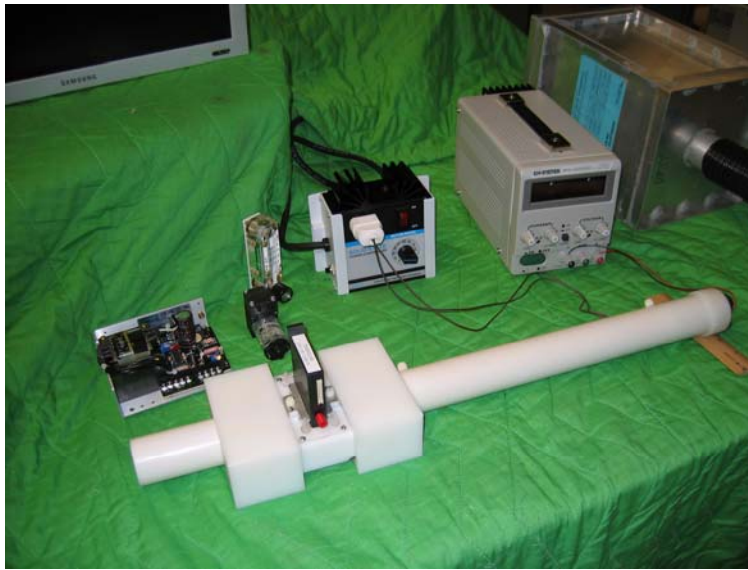


Fig. 9. An image of a droplet conditioning module for rapidly drying the droplets generated by inkjet printheads to produce the final aerosol particles. The unit is shown with a short printhead plate, to accept a single printhead. The long end of the tube houses the intake fan and hot air duct, and the dried particles exit from the short tube.

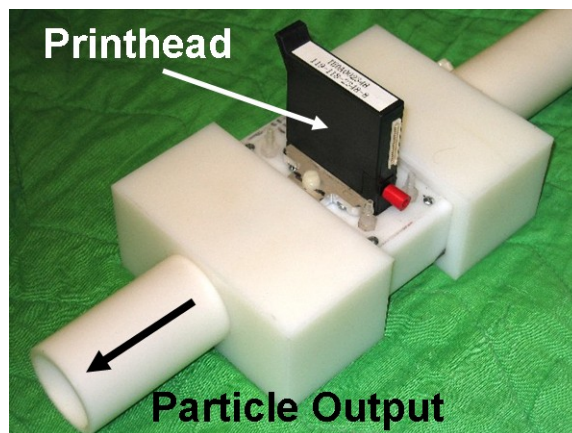


Fig. 10. A closeup of the output end of the module, showing the configuration of the printhead and the particle output duct. The printhead plate can be replaced with a version accommodating up to 4 printheads.

Four parallel cutouts 3/8 inches deep are machined into the top plate to accommodate up to four printheads. The printheads fire droplets through a 1/8 inch wide slot in the bottom of each cutout, into the hot air flowing underneath. The printheads are docked into close fitting U-shaped structures fastened above the cutouts, and held in place by the side pressure of a single screw. With this mounting assembly any printhead can be quickly removed for servicing and easily replaced again.

Windows, 1 inch high and 4 inches long, are provided in the left and right duct sides at the upstream end where the printheads are located. Each window is a double pane of 1/8 inch thick borosilicate glass, with one pane set into the inside wall of the Teflon side piece and one pane on the outside wall. The purpose is to allow observation of the droplets as they are ejected from the printhead so that problems such as dripping nozzles can be immediately corrected.

Each end of the duct mounts into a Teflon block in which flow transition takes place. On the downstream side the aerosol-laden air is directed into a 1 inch diameter tube from which it emerges ready to be coupled into an experiment. The upstream block connects the duct to the output of a hot air oven.

The hot air oven components are contained within a tube 2 1/2 inches in diameter and 15 inches long, which is inserted into the Teflon block that couples to the duct. At the opposite end of the tube is a 60 mm fan, Micronel D601L-024KA-3, that pushes air through the entire system. The fan motor is a brushless dc type rated for 24 volts; the flow can be reduced by reducing the voltage.

Between the fan and the duct lies a heater cartridge capable of quickly raising the temperature of air flowing through it. Within the cartridge is a thin walled brass tube, 1-3/8 inches diameter and 12 inches long. It is wrapped with fibrox insulated heat tape, 1/2 inch wide and 4 ft long, capable at full power of 900 °F. The interior of the brass tube is tightly packed with smaller (3/16 inch diameter) aluminum tubes. Their purpose is to use direct conduction to heat the entire interior of the brass tube rather than just its surface. The heat tape winding is thermally insulated with a woven fiberglass sleeve and an outer layer of ceramic tape (non-adhesive). That structure is inserted into a 2 inch diameter stainless steel tube with screened end caps that center the brass tube and pass electrical connection to the heat tape.

This heater cartridge is itself insulated with fiberglass and ceramic tape and inserted into the 2 1/2 inch stainless steel tube, between the fan and the duct. The graphs below display measured performance of the hot air oven. Fig. 11 shows air flow through the system as a function of fan voltage. Fig. 12 shows the temperature of air exiting the oven as a function of setting on the heat tape power controller and for several air flow rates.

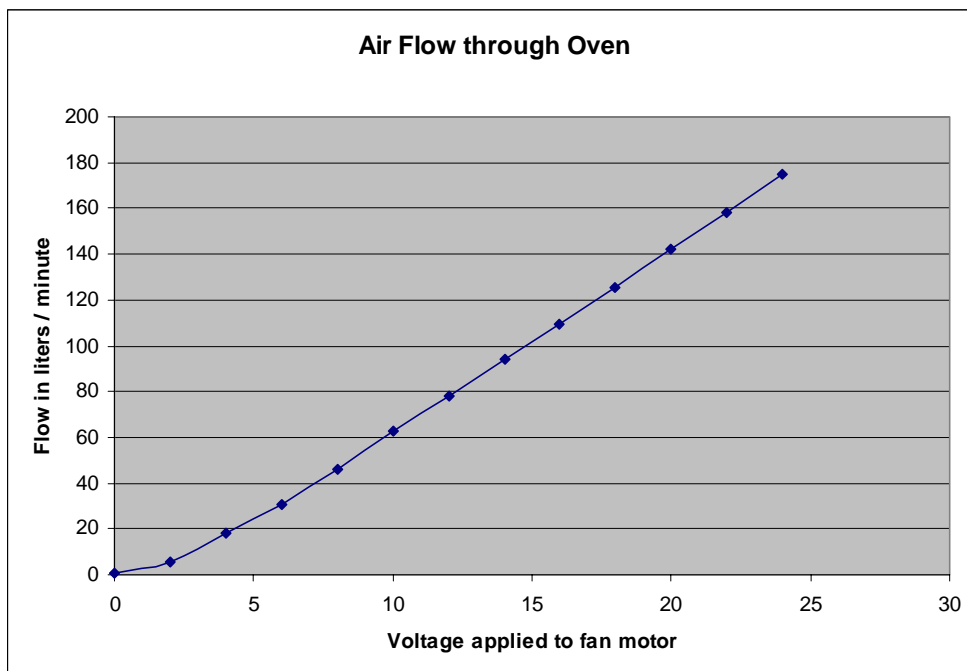


Fig. 11. Chart showing the air flow through the oven tube of the droplet conditioning module for various supply voltages to the fan motor.

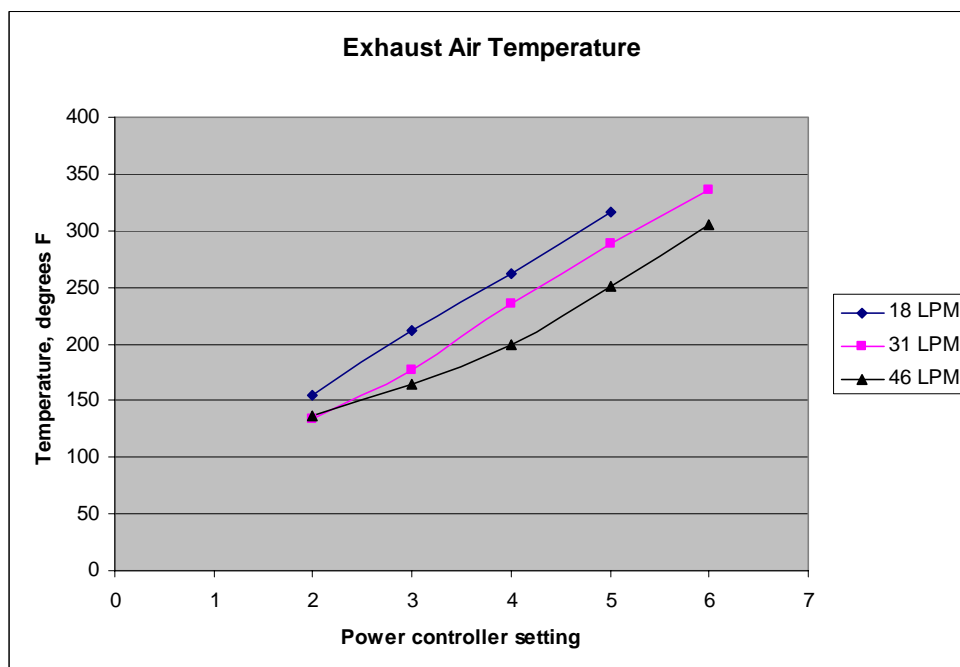


Fig. 12. Chart showing the exhaust air temperature from the oven tube for different heater power settings and air flow rates.

All the droplet drying module components are inserted into one another and fastened to a single Plexiglas platform for convenience. Also on the platform is the power controller for temperature control in the oven, and space is reserved to mount the liquid flow devices that feed the printheads. A two channel thermocouple is included to monitor air temperature (through small holes in the Teflon transition blocks) at the duct entrance and exit.

During the course of the development program, there were upgrades to the droplet conditioning design in areas for which performance or ease of use could be improved, based on the results of development testing. Specifically, a quick release printhead mount replaced a screw-down mounting clamp that proved cumbersome in practice. Windows are now provided because we felt a need to see the printhead performance in real time rather than inferring its condition from APS data. The original module relied heavily on delrin for its construction, even in its oven, and so a delicate balance had to be reached between achieving adequate temperature in the duct while not melting other plastic parts. The final version can sustain, without damage, temperatures much higher than should ever be required to dry aerosols.

3.4. Additional Hardware Components

Some additional components were developed subsequent to the core inkjet system tests, in order to boost system performance and user friendliness. The gravity-based liquid feed pressure control configuration was replaced with a specially built electronically controlled, closed-loop pressure control subsystem. This system consists of a fluid reservoir, a miniature air pump, a pressure transducer, and associated tubing and interconnects. The pressure subsystem is operated through the same graphical user interface (GUI) used to manage the other aerosol generation functions, as shown in Fig. 8. The fluid supply tasks include both filling the printhead with solution at the start of a test, and supplying it with a steady pressure to ensure sufficient supply of solution during operations. The pressure control subsystem allows the user to manage both these processes controllably. The pressure is controlled relative to ambient, and the latest modifications to the system incorporate two identical transducers in order to correct for variations in ambient pressure.

Other additional components include a pressure relief valve, a compact 24V power supply, a 5V regulated power converter, and additional electronics to allow interface to the control computer via a data acquisition (DAQ) card (National Instruments).

3.5. Aerosol Testing

The droplet conditioning module was integrated with the core inkjet system, and underwent aerosol generation tests at Lawrence Livermore National Laboratory. The system as tested incorporated the droplet conditioning module, a KM256 inkjet printhead and associated EB-100 electronics, the closed loop liquid pressure control system, off-board power supplies, and a late version of the GUI control software.

The Digital Aerosol Generator (DAG) was located outside the flow-through, tunnel type aerosol chamber, with the particle exit duct passing through a hole in the side of the chamber to allow the release of aerosol into the chamber interior. The system under test is shown in Fig. 13. The chamber was equipped with an aerodynamic particle size measurement system (APS), used to capture size histograms of the distribution of particles in the interior.

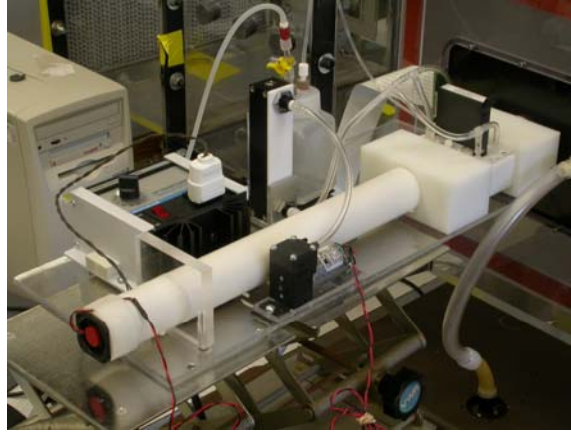


Fig. 13. The DAG system undergoing aerosol generation tests at the aerosol test facility at LLNL. The side of the aerosol chamber can be seen at top right. This build incorporated the single-printhead plate.

3.5.1. Size distribution measurements

In these tests, the viscosity of aqueous solutions was varied by using solutions of different concentrations of glycerine in de-ionized water. The goal was to verify that the results of the core inkjet tests hold for the complete system, including the droplet conditioning module. The earlier tests had indicated through stroboscopic imagery that the printheads yield a very monodisperse population of droplets, all being essentially identical in size, and that the dynamics of droplet generation can be controlled by adjusting pulse width in order to ensure the generation of single droplets free of satellites. After the droplet conditioning tube had reached operating temperature, droplets were generated and size distribution histograms of the air in the chamber were captured for different operating conditions.

Typical results are illustrated in Fig. 14. These aerosols were generated using 0.3% glycerine solution, with a viscosity very close to 1.0. At left is the size distribution of dried aerosol particles resulting from a pulsewidth of 3 μs . By referring to Fig. 6, we can see that this condition is expected to yield satellite droplets. The sharply bimodal distribution in the APS data confirms the production of satellites. At right is the same case, except that the pulsewidth is adjusted to 2.5 μs . Here, the data of Fig. 6 predict the generation of single droplets, and the APS data verifies this by showing a single, highly monodisperse peak in the size spectrum.

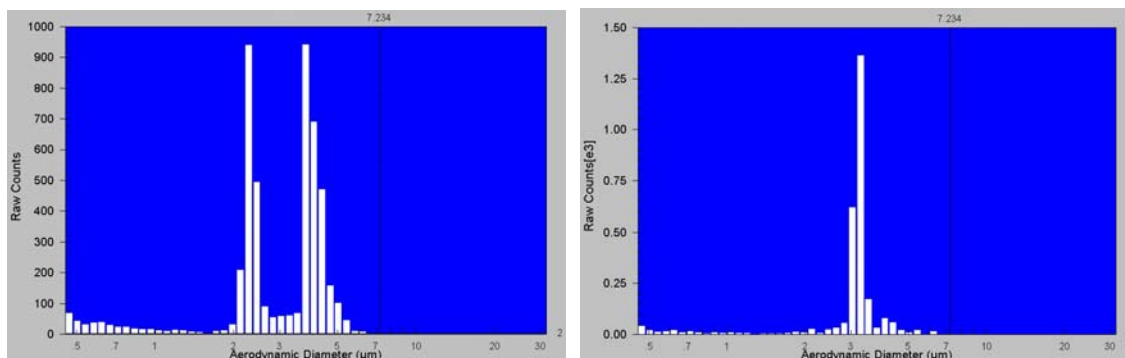


Fig. 14. Aerodynamic particle sizing (APS) histograms for dried glycerine test aerosols generated by the DAG unit. The source solution was 0.3% glycerine. At a pulse width of 3 μs , at left, satellite particles are detected. These are eliminated when the pulse width is adjusted to 2.5 μs , at right. Note the highly uniform size distribution for the single droplet generation.

3.5.2. Particle size control

As with the IJAG, control over the dried size of the aerosol particles generated by the DAG is provided by the ability to control the concentration of the dissolved solute in the aqueous solution supplied to the printheads. The less solute, the smaller the final aerosol particles. The relationship of solute concentration to particle size was investigated experimentally by generating monodisperse aerosol particles from different glycerine solutions and measuring the sizes of the resulting particles using the APS. The results are shown in Fig. 15. As expected, lower solute concentrations lead to smaller particle sizes, and for relatively non-volatile solutes such as glycerine the dried size can be calculated from the volumetric ratio of solute to solvent, as indicated by the dashed line.

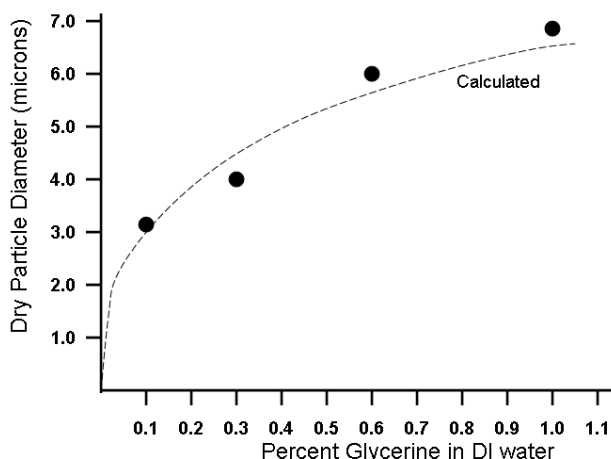


Fig. 15. A chart showing the measured values of dried glycerine aerosol particle diameter for source solutions of different concentration. The calculated relationship (dashed line) is based on simple volume ratios, assuming a standard 30 μm droplet size.

3.5.3. Biological slurries and spore simulants

One of the key applications for the DAG is the generation of controlled aerosols from solutions containing discrete biological particles, such as bacterial spores. In the aerosol chamber environment, dried aerosols were generated from two representative types of solutions, and the resulting particles were measured and captured on filter paper for subsequent imaging. This capture was performed by mating the suction inlet of the APS to a filter holder containing a replaceable disk of 0.8 μm pore size polymer membrane filter paper (Millipore), and positioning the modified inlet close to the exit duct of the DAG so that particles would be attracted to, and adhere to, the filter paper for later examination.

The first was a solution of 1 μm carboxylated polystyrene microspheres (Polysciences Inc., Warrenton, PA). These served as a low cost simulant for bacterial spore slurry, and could be obtained in high purity and large volume. As in the case of the particle size control tests performed with glycerine solutions, the lower the concentration of polystyrene beads in the solution, the smaller the resulting dried particles. For low concentrations, the highest count of aerosol particles was composed of single polystyrene microbeads. At higher concentrations, clusters became evident, as shown in Fig. 16, scanning electron micrographs of the particles captured on filter paper.

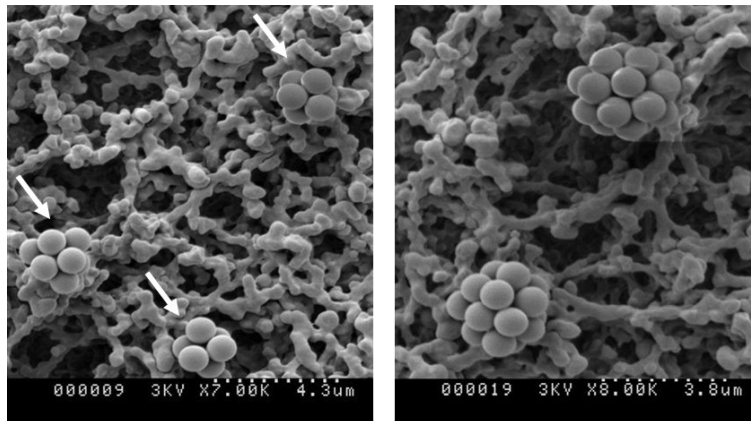


Fig. 16. SEM images of polystyrene microbead clusters produced by the DAG from microbead suspensions of different concentration. Upon drying, the water in each droplet evaporates, leaving the particles behind as a packed cluster. At left, three clusters are indicated. The larger clusters at right were produced using a somewhat more concentrated solution. The complex material in the background is the surface of the filter paper.

Similar tests were performed with a suspension of *Bacillus globigii* (Bg) spores, a common harmless simulant for *Bacillus anthracis* (anthrax). In contrast to the monodisperse size distributions seen with purer solutions, the Bg slurry produced a broader distribution of dry particle sizes, with a tail at the small end of the distribution. According to personnel at the aerosol test facility, this is typical of aerosols from biological slurries, as they contain not only spores but fragments of spores, various debris from the fermentation and sporulation processing, dissolved proteins and other nonvolatile chemical species. Electron microscopy of the filter paper revealed clusters similar to those seen with the polystyrene microspheres. Fig. 17 shows such a cluster.

The Bg spore concentration in the source solution was $6.5 \times 10^9/\text{ml}$, suggesting a concentration of 90-100 spores per $30\ \mu\text{m}$ diameter (15 pl) droplet, a number in accordance with the appearance of the clusters.

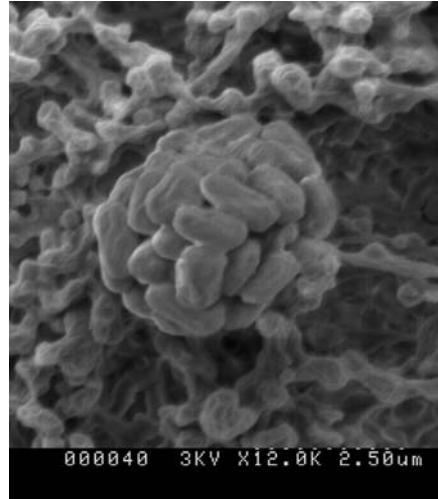


Fig. 17. A cluster of Bg spores produced from a suspension of spores in water. Calculations predict 90-100 spores in the cluster, based on the approximate concentration of the spore suspension.

3.5.4. Programmed Sequence Operation

One of the important features of the Digital Aerosol Generator is the ability to accept pre-programmed aerosol generation sequences and execute them automatically as part of a dynamic aerosol test procedure. The last major development test was an aerosol chamber test to verify the operation of this feature. The intake tube for the APS was located at the exit of the DAG particle exit duct, in order to capture a consistent fraction of the total number of particles exiting the generator. A pre-programmed sequence was run, using the features of the custom software built for this purpose. The sequence consisted of a series of timesteps with different printhead frequencies. The APS sampled repeatedly for fixed integration times over the course of the test, and the total counts for each sample time were compared with the printhead frequencies commanded by the pre-programmed sequence. The test was repeated several times.

Results are shown in Fig. 18. Note that the units for the programmed steps in printhead frequency and the sampled number of particles are different. The two are directly proportional to each other throughout the programmed sequence, demonstrating that the programmed sequence feature operates as intended.

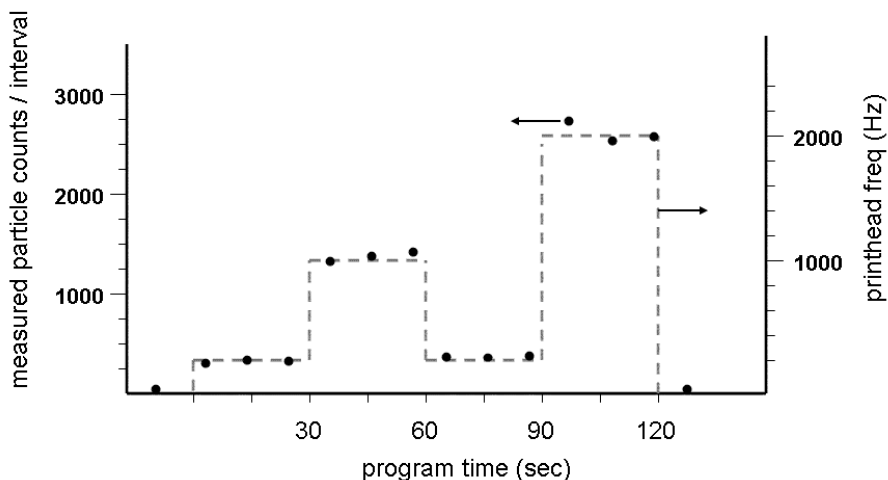


Fig. 18. Data from aerosol chamber testing of the programmed sequence mode of the Digital Aerosol Generator. The right scale and dashed line indicate the sequence of printhead frequency steps programmed into the DAG control. The data points and left scale correspond to the integrated particle counts for fixed (5 second) samples taken at the exit duct.

4. Programmatics

The schedule for the project was 12 months, and the project budget was \$541K, with \$115K of this total allocated to ECBC for the droplet conditioning module development and the balance allocated to LLNL. The project satisfied all the requirements of the statement of work within the provided schedule and budget.

The one potential deviation from the project baseline came about due to an extensive and unexpected 8-month delay in the transfer of the \$115K from LLNL to ECBC. Various administrative issues at the LLNL, Department of Energy, Department of Defense, and ECBC levels resulted in a delay much longer than had been anticipated, imposing a late start on Task 2, the development of the droplet conditioning module. Nonetheless, the project team was able to work around the delay and accomplish all the project goals. If the droplet conditioning modules had been available sooner, the extra time would have been spent in optional explorations of the aerosolization behavior of additional liquid solutions, and attaining additional aerosol test data beyond that required.

Due to careful control of spending, management reserve was available to fund the fabrication of a second complete set of Digital Aerosol Generator components at the end of the project. Therefore, instead of one DAG available at ECBC for follow-on work, two systems are available, one at ECBC and another at LLNL.

5. Summary

The project CA06TAS446 “Engineered Aerosol Production for Laboratory Scale Chemical / Biological Test and Evaluation” was a brief, 12-month project to prototype and test a novel advanced aerosol generator based on commercial off-the-shelf array

inkjet printing technology. The project achieved all objectives in the statement of work within the allocated schedule and budget.

The results of testing demonstrate that the Digital Aerosol Generator (DAG) can produce high fluxes of monodisperse aerosol particles (over 2.5 million per second per printhead). The dynamics of the droplet generation process can be controlled electronically to ensure the production of monodisperse droplets from the printheads over a wide range of viscosities. Using the KM256 printhead, the droplet size is 30 μm diameter (15 pl volume), and the size of the dry aerosol particles is determined by the concentration of solute within the solution. A droplet conditioning module was developed to rapidly dry the inkjet droplets into dry aerosol particles, and interface with aerosol chamber equipment. The system includes automated liquid feed pressure control, and can accommodate up to 4 printheads simultaneously. System operations are controlled via custom PC-based software with an easy-to-use graphical user interface, including the capability to run user-defined pre-programmed test sequences. The DAG has demonstrated its ability to generate dry aerosols from particle slurries of polymer microbeads and bacterial spores. While it should still be considered a developmental prototype, the Digital Aerosol Generator has demonstrated capabilities that make it attractive for high performance, dynamic chemical / biological aerosol chamber testing.

Appendix: System software and operation

This section provides a concise overview of the operation of the Digital Aerosol Generator (DAG), with emphasis on the control software. Because the DAG remains at the prototype stage of development, some details may continue to evolve over time, and some aspects of the operation may not be as robust as would be expected for a fully developed production unit. The information provided in this section serves as a useful introduction to key system operations. It is not intended to serve as full production documentation.

Software and Installation

The system control software is built on LabView 8.0 (National Instruments). It is necessary to install LabView in order to operate the DAG control software. Future versions may be available in the form of an executable file that does not require LabView.

The main DAG control .vi file, **Inkjet Aerosol_Event_State_Machine_V1.vi**, and the other .vi files provided must be installed in a directory, and this directory added to the path in LabView.

The control software is optimized for the National Instruments data acquisition (DAQ) hardware currently used with the system. If different computer interface hardware is to be used, modifications to the LabView code may be required.

Hardware Configuration

The setup of the DAG requires the following components:

- Digital Aerosol Generator, including on-board power supplies and fluid delivery subsystem
- Printheads (up to 4), either KM256 or KM512
- EB-100 electronics unit
- PC with LabView and DAG control .vi files installed and appropriate DAQ hardware
- Serial cable to connect PC to EB-100
- Ribbon cables to connect EB-100 to printhead(s)

The DAG should be oriented in a horizontal position, with the printheads at the top of the unit. The components should be connected with the appropriate cables. The liquid feed tubing from the fluid delivery subsystem should be inserted into the fluid input port for the printhead(s).

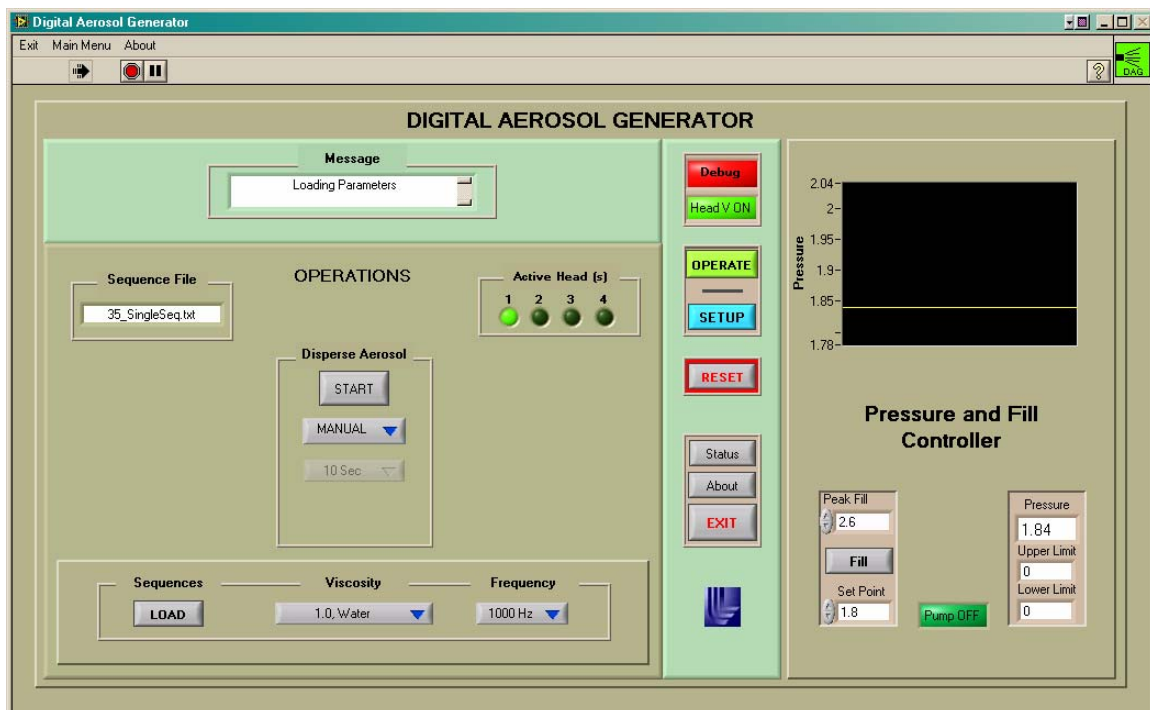
Startup

1. Turn on the droplet conditioning module and allow the oven to heat to a stable temperature. Ensure the air flow fan is operating.
2. Boot the PC up, and open LabView.
3. Load the main DAG control file but do not Start the .vi.
4. Ensure that the LVDS switch at the back of the EB-100 unit is in the ON position.
5. Turn on the EB-100 using the switch on the front of the unit.
6. Start the main .vi within LabView.

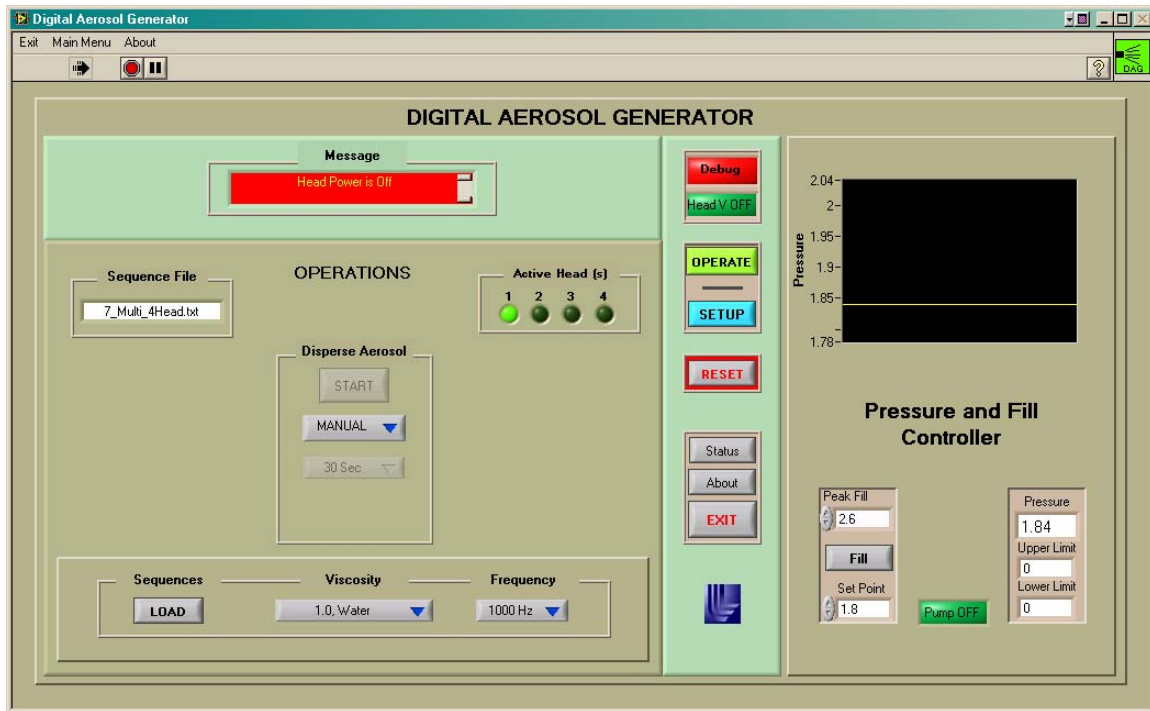
If the EB-100 unit is not recognized, or a “check power / connections” error is displayed, turn the EB-100 unit off, close the .vi, and resume the startup process at step 3. If this does not resolve the problem, exit the software, restart the PC and begin at step 1.

Front Panel

This image shows the front panel of the DAG graphical user interface (GUI).



The basic aerosol generation operations can be accessed from this panel. For simplicity, these overview instructions will assume that a single printhead is installed. If printheads are to be replaced (or added) during operation, the “Head V ON” button should be toggled to the OFF state, as shown in the following image. This disconnects the power to the printheads and allows them to be disconnected from the system without damage. When a new printhead is installed, toggle the button back to enable normal operations.



Printhead Filling

This procedure is needed to load an empty printhead with solution prior to aerosol generation. Check that the liquid supply reservoir has been loaded with the desired solution for aerosolization, and that all fluid lines are connected. Using the right side of the front panel, set the Peak Fill value to a value slightly above the set point (atmospheric pressure) and toggle the “Pump OFF” button to “Pump ON” to enable the miniature air pump to become active. Note that the pressure values are given in voltages from the pressure transducer: atmospheric pressure is approximately 1.8 volts, and the voltage increases at 20 mV/kPa. Press the “Fill” button, and the pressure in the reservoir will increase, as indicated on the Pressure chart. Slowly increase the Peak Fill value while observing the fluid line to the printhead. Typical values for filling the KM256 printheads with low-viscosity aqueous solutions are about 0.4-0.8 V above atmosphere.

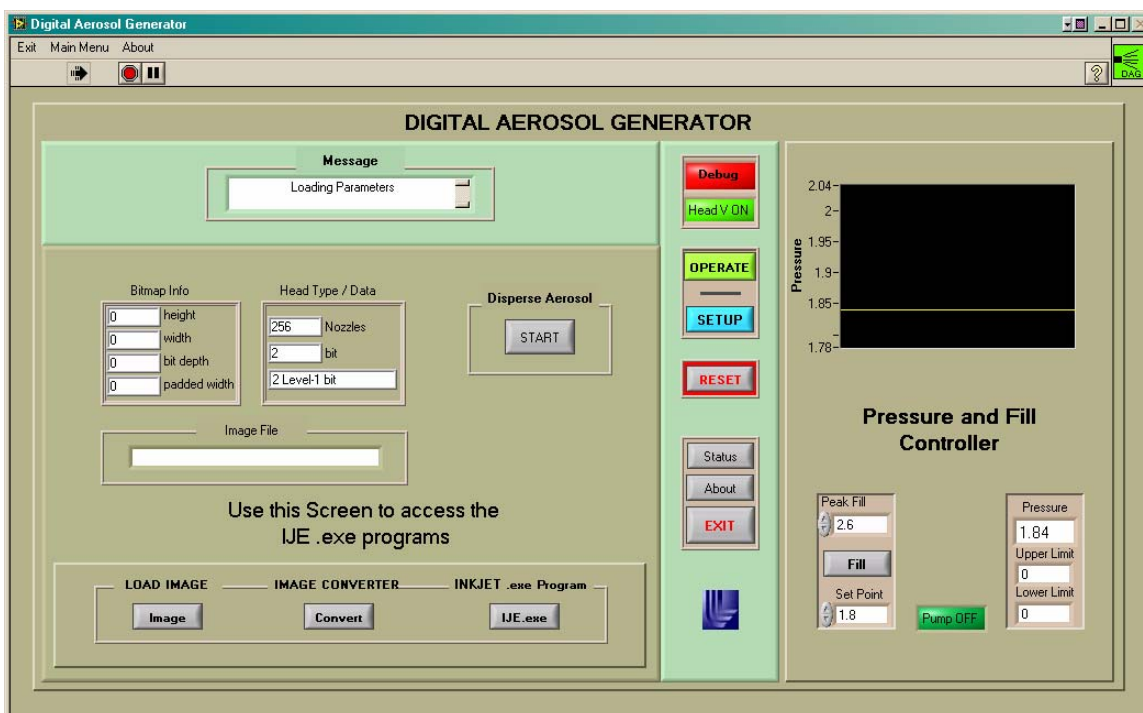
Once the solution has risen through the fluid line and into the printhead, the “Pump ON” button can be toggled back to “OFF.” Release the pressure in the reservoir and wipe any excess liquid from the print nozzles.

During operation, the Peak Fill pressure should be set to slightly above atmospheric pressure. Optimal values depend on the viscosity of the liquid and may need to be adjusted periodically. Values of 0.02 to 0.20 V above the atmospheric reading are typical. The objective is to use a pressure that is high enough to ensure a good supply of liquid to the printhead during aerosol generation, and low enough to prevent the leakage and dripping of liquid from the inkjet nozzles.

Aerosol Generation

On the front panel, choose an appropriate viscosity value from the pulldown menu at bottom. This choice sets the waveform parameters for optimal single droplet generation. Then choose the desired printhead frequency from the pulldown menu immediate adjacent. This determines the rate at which aerosol particles will be generated from each of the nozzles (256 in the case of the KM256 printhead).

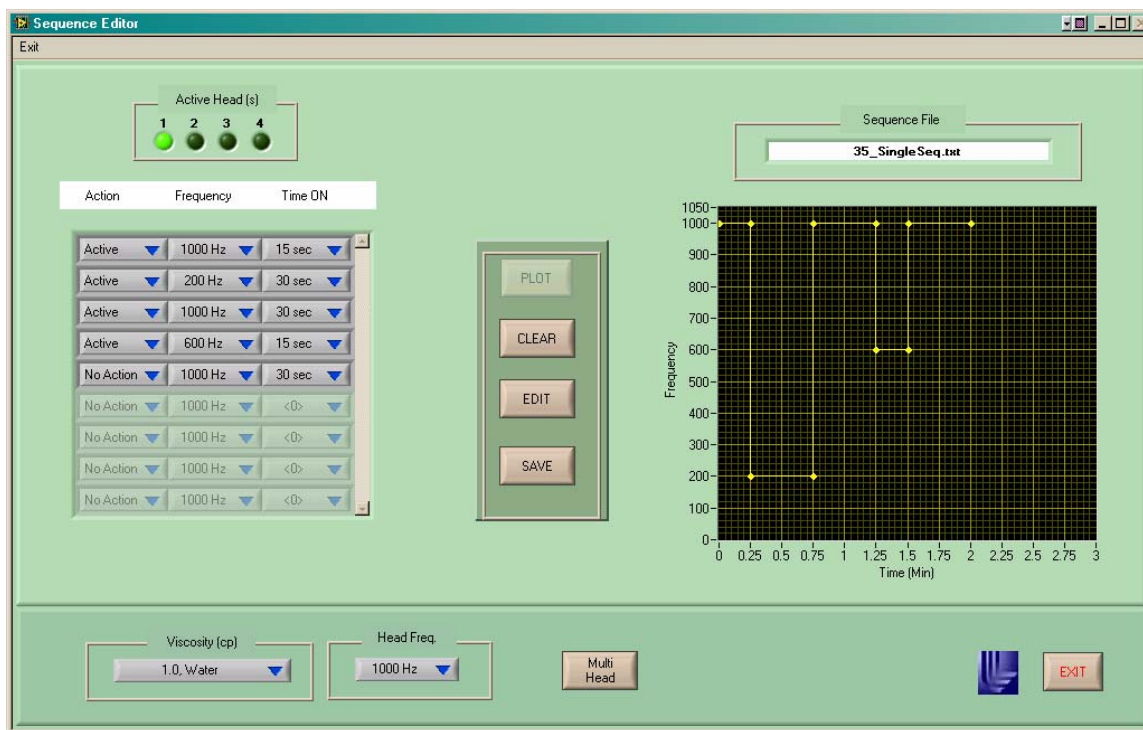
If a different printhead is desired, this can be specified on the Setup panel, accessed by pressing the “SETUP” button on the front panel. The Setup panel is shown below.



This screen allows for additional options that are outside the scope of this overview. An Advanced Setup panel is also available, allowing for manual control over head voltages, DRC waveform parameters, and other features.

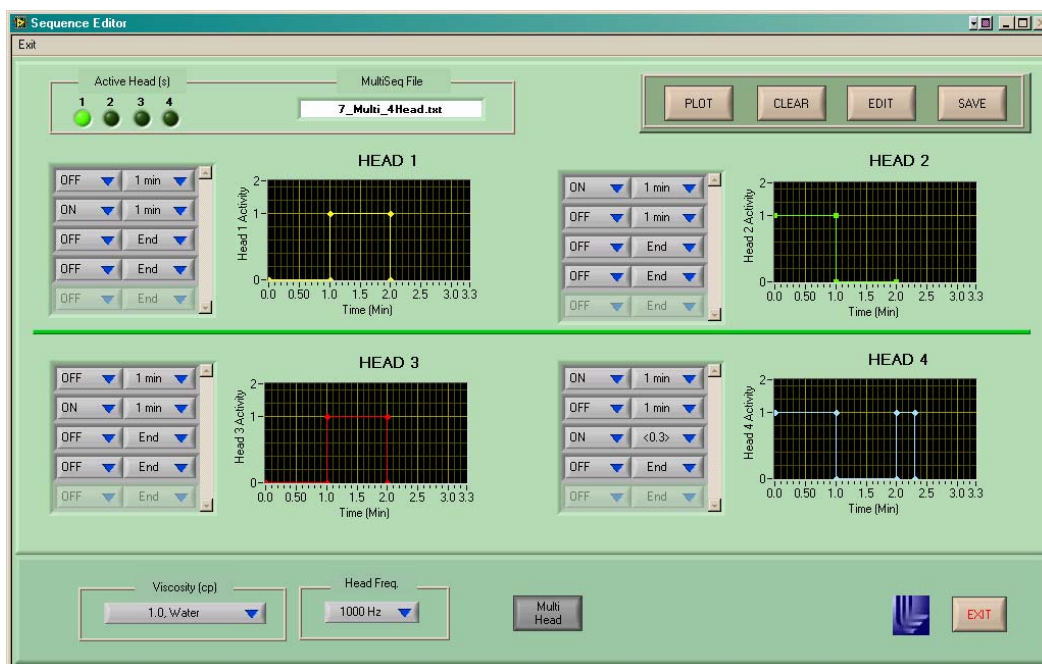
Returning to basic operations, under the “Dispense Aerosol” section of the front panel, any of three operating modes can be chosen: Manual, Timer, or Sequence. If Manual operation is chosen, aerosol generation can be started by pressing the “OPERATE” button in the center panel. Toggle this button to start and stop aerosol generation. The Timer option allows for aerosol generation to proceed for fixed periods of time before stopping automatically.

The Sequence option allows the use of programmed stepwise aerosol generation sequences, that may be saved and re-used. Selecting the Sequence option allows an existing sequence to be loaded using the Load button and sequence file name box. A new sequence can be created using the Sequence editor screen, shown below.



A sequence can be easily built using this feature. To build each step in the sequence, select “Active” in the Action field, then choose the desired printhead frequency and duration in the other two fields. The final step should have “No Action” specified in the Action field. The plot will illustrate the resulting sequence.

Sequences can be built for multiple printheads. To do this, select the “Multi Head” button at the bottom. Up to four heads can be used in a given sequence.



Note that the current version of the DAG software restricts multiple printheads to a common printhead frequency. This is a result of firmware in the EB-100 electronics, designed to keep the printheads synchronized during four-color image printing.

Storage

Between uses, the printheads should be flushed of solution and, if need be, decontaminated. When stored, they should be filled with a nonvolatile storage solution (such as the ethylene glycol / propylene glycol / water solution provided for this purpose by Konica Minolta). This prevents the drying of solution inside the printheads, which may result in the deposition of residue and clogging of the inkjet nozzles.